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Dated 2 March 1999

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1. Your reference

C324/G

2. Patent application number
(The Patent Office will fill in this part)

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Michael COLE
Poplar Farm
Molesford
Woodbridge
Suffolk
IP13 0AL
United Kingdom

Patents ADP number *(if you know it)*

0642317200 1

If the applicant is a corporate body, give the country/state of its incorporation

4. Title of the invention

Control of weight during evaporation of samples

5. Name of your agent *(if you have one)*

Keith W Nash & Co

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

90-92 Regent Street
Cambridge
CB2 1DP

Patents ADP number *(if you know it)*

1206001

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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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Number of earlier application	Date of filing *(day / month / year)*

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Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature Keith W Nash & Co, Agents Date 11.11.98

12. Name and daytime telephone number of person to contact in the United Kingdom: Mr Nash (01223) 355477

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C324/G

Title: Control of weight during evaporation of samples

Field of Invention

This invention relates to a method of and apparatus for controlling the weight of samples dissolved or suspended in a liquid while they are evaporating in a vacuum. It is particularly applicable to samples in centrifugal evaporators.

Background to the Invention

Samples to be evaporated in centrifugal evaporators are usually held in glass or plastic tubes or, sometimes, in a large number of small wells in plastic blocks. The sample holders are mounted upon a rotating assembly and spun at relatively high speed so that a considerable centrifugal force is applied to them in an outward direction, which forces the liquid to the lower part of the sample tubes and prevents any frothing or spitting of the liquid out of the sample tubes when a vacuum is applied. The spinning samples are held in a vacuum-tight chamber (referred to herein as a "chamber") which is connected to a vacuum pumping device.

Centrifugal evaporators of this type are well known and many types are available commercially. One problem from which such evaporators suffer, is that it is very difficult to obtain a desired continuous read-out of the weight of the sample in the holders as the liquid is being evaporated, since the holders are being spun at a high speed, typically at about 1400 r.p.m. The possibility has been considered of continuously weighing the whole evaporator during spinning. However, this involves measuring a total weight of the order of 50 kg to an accuracy of about 1 gm, which is a very demanding task.

Another problem arises when evaporation needs to take place simultaneously for different

solvents, or solvent mixtures of differing compositions, in which the samples are dissolved or suspended. In this situation those samples which are held in the more volatile solvents or mixtures will evaporate faster than the ones held in the less volatile solvents, and this can lead to an excessive imbalance in the rotating assembly, and consequent unwanted vibrations. This would also mitigate against the possibility of weighing the whole evaporator.

In most centrifugal evaporator machines such unwanted vibrations are arranged to trip an out-of-balance sensor to thereby stop the machine, but in machines without a sensor the vibrations can cause damage to the machine and even to the samples. Sometimes the vibration problem can be overcome by careful loading of the evaporator, or by stopping the process from time to time and rebalancing the load by adding liquid to empty samples or by rearranging the samples in the rotating assembly. Both these methods are tedious and time consuming.

It is an object of the present invention to enable the weight of a sample in a centrifugal evaporator to be continuously and accurately measured during evaporation.

It is another object of the invention to enable the operation of a centrifugal evaporator to continue despite a considerable imbalance of forces.

Summary of the invention

According to one aspect of the invention there is provided a method of weighing a sample dissolved or suspended in a liquid during evaporation in a chamber of a centrifugal evaporator, comprising the steps of attaching a force measuring transducer between a sample holder and a rotor, obtaining force signals from the transducer while the rotor is spinning, and converting the signals into corresponding weights by taking account of a factor for the centrifugal force exerted on the sample holder.

The invention also extends to apparatus for measuring the weight of a sample as aforesaid, comprising a vacuum chamber, a sample holder connected to a rotor therein, a force measuring transducer attached between the holder and the rotor, and means for transmitting signals from the transducer to a reception means outside the chamber, and means for converting the signal to a value proportional to the weight of the sample holder.

The weight of the sample can then be continuously calculated by taking account of the centrifugal force and deducting the known weight of the holder.

The transducer is preferably a load cell; alternatively a strain gauge or position sensor may be utilised.

According to another aspect of the invention in the processing of samples in a centrifugal evaporator in which the samples are dissolved or suspended in liquids of differing volatility, any imbalance caused during spinning of the rotor and resulting in unwanted vibration is at least partially compensated for by associating with the rotor an automatic balancing unit.

Preferably the said unit comprises a bearing raceway incorporating a plurality of rolling elements such as ball bearings which incompletely circumferentially occupy a raceway, and which in use automatically distribute themselves to counteract the said imbalance.

Only limited space is available within apparatus as described herein for laboratory use and the like, and according to a further aspect of the present invention, the rolling elements are constructed from a dense material such as Tungsten or depleted Uranium. This allows the overall size of the raceway to be reduced both in depth and diameter, due to the increased mass of the rolling elements obtained by using high density materials therefor.

Brief description of the drawings

The invention will now be described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 is a schematic side view of a centrifugal evaporation system incorporating a force measuring transducer in accordance with the invention; and

Figure 2 is a perspective view of a dissembled automatic balancing unit associated with the rotor of Figure 1.

Detailed description

Referring first to Figure 1, this depicts a complete centrifugal evaporating system which is more fully described in copending Application No. 9803684.1 (ref: C311/G). The subject matter of said Application is therefore hereby entirely incorporated by reference into the present Application.

Sample holders in the form of blocks 4 are connected to pivots 13 such that the samples are held vertical during loading into the chamber 14. When the rotor shaft 5 is rotated the blocks swing out to a horizontal position due to centrifugal force.
Attached between each blocks and the shaft is a load cell 17 which records the horizontal force on the block which, when the rotor is spinning, will be proportional to the weight of the sample plus the sample holding assembly. Since the latter is constant the sample weight can readily be obtained. Of course, the apparent weight will be exaggerated by a factor due to the centrifugal force, but this factor will remain constant for a given rotor speed.

The power supply for the load cell may be provided in the same way as that for the direct temperature measurement described in the above mentioned Application No. 9803684.1. The weight signal is processed and transmitted to a receiver and decoder outside the

chamber in a similar manner but via a separate channel.

In this way experiments have shown that one can measure weights of samples in holders weighing up to 1200 gm to an accuracy of better than 1 gm. This measurement allows accurate assessment of the correct moment at which to switch off sample heat, and of the point at which the samples are completely dry.

Figure 2 shows a proprietary automatic balancing unit which is preferably fitted to the rotor shaft 5 as close as possible to the sample blocks 4. Here vibration caused by rotor imbalance is likely to occur, when solvents of different volatility are used for the samples.

The unit may be the Auto-Balancing unit produced by the bearing manufacturing company SKF.

As shown in Figure 2, the unit comprises inner and outer raceways 20 and 22 between which a number of loose ball bearings 24 are freely movable. The ball bearings distribute themselves automatically to counteract the imbalance in the rotor shaft 5.

Balancing devices which can be used to reduce out-of-balance forces in weighing apparatus of the type described herein are described in WO 98/01733, and in accordance with the preferred aspect of the invention, the ball bearings are of a material having a density higher than that of steel, such as Tungsten or depleted Uranium.

CENTRIFUGAL EVAPORATION SYSTEM

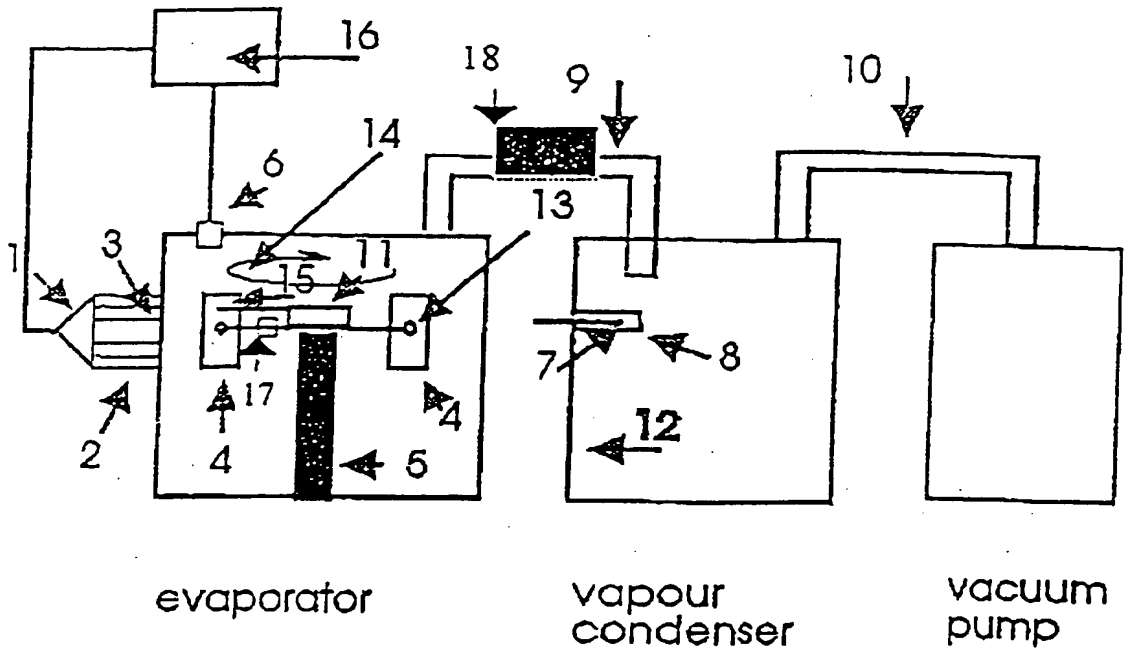

FIGURE 1

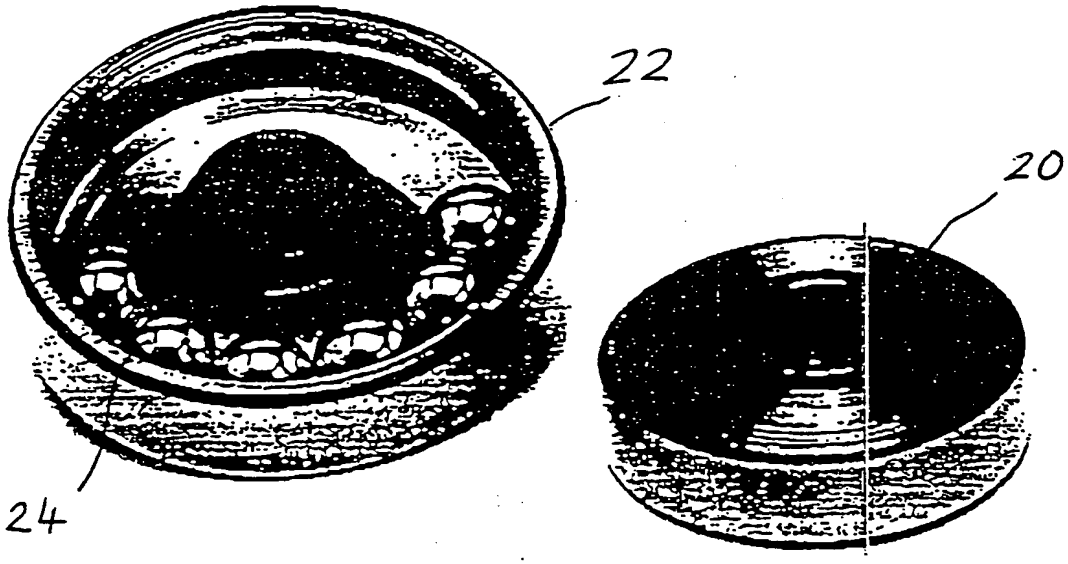

FIGURE 2